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CHARACTERIZATION OF HYBRIDIZED CONDUCTIVE COMPOSITES

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Injection moldable thermoplastics filled with various hybrid conductive reinforcements have been investigated for their physical/mechanical properties. The thermoplastics studied include polyphenylene sulfide (PPS), polyether-etherketone (PEEK) and liquid crystal polymers (LCP's). The reinforcements included various combinations and concentrations of graphite (Gr) fibers, stainless steel (SS) fibers, nickel flakes (NiFlk) and nickel plated graphite (NiGr) fibers. Tensile, flexural, and drop weight impact tests were performed. Fracture morphology of tensile specimens was investigated using scanning electron microscopy (SEM). The distribution of fibers and flakes was observed using photomicroscopy. Electromagnetic interference (EMI) shielding effectiveness was determined in the range of 15 MHz to 1 GHz. Water absorption, density and electrical resistivity properties were also determined. Differential scanning calorimetry (DSC) was

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used to determine thermal properties. Four PPS composites provided at least 80 dB of shielding at 80, 100, 150, 200 and 300 MHz respectively. The lowest electrical resistivity value measured was 0.60 ohm-cm for PPS/30%NiGr/10%SS. Optical microscopy revealed that PEEK composites with similar loadings to that of PPS composites had lower void contents. Reinforced PEEK performed better mechanically than PPS composites. Composite densities ranged from 1.98 g/cm³ for PPS/30%NiGr/20%NiFlk to 1.47g/cm³ for LCP300/30%Gr.

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INTRODUCTION

Considerable effort to replace primary and secondary airframe structures with advanced composite materials has resulted in significant weight savings and improved aircraft performance. However, non-structural, electromagnetic interference (EMI) critical aircraft components have not been targeted frequently as composite replacement items. Aluminum avionics enclosures and connectors often require costly secondary machining steps and frequent corrosion maintenance actions. Injection moldable thermoplastics containing conductive reinforcements have the potential to provide adequate EMI shielding, better corrosion resistance, and considerable cost and weight savings.

Previous efforts have characterized the effect of a single conductive reinforcement in a thermoplastic matrix [1,2]. The work presented in this report exploits a hybridized reinforcement approach to enhance the EMI attenuation characteristics of composites. Thermoplastics filled with nickel plated graphite fibers in tandem with another fiber or flake material were studied. The electrical, mechanical, physical, electrochemical and thermal properties of these materials were evaluated.

MATERIALS

All of the resin materials tested were injection moldable thermoplastics. Composite and unfilled resin samples composed of polyphenylene sulfide (PPS) and polyether-etherketone (PEEK) [3] were provided by RTP Company (Winona, MN). Two liquid crystal polymers (LCP's) [4] introduced under the trade name of XYDAR™ by Dartco Manufacturing Inc. (Paramus, NJ) were also investigated. Both the XYDAR SRT-300 and XYDAR SRT-500 grade LCP's are aromatic copolyesters composed of para-oxybenzoyl repeat units and oxybiphenylene terephthaloyl repeat units. Various concentrations and combinations of graphite (Gr) fibers, nickel plated graphite (NiGr) fibers, type 304 stainless steel (SS) fibers and nickel flakes (NiFlk) were tested in the PPS and PEEK matrices. The compositions of the reinforcements in PPS were chosen using an extreme vertices formulation design. The LCP samples were tested with only Gr fibers as the reinforcement.

The mold temperature and injection pressure of the PPS and PEEK composites were 163°C and 138 MPa, respectively. The temperatures of the molten PEEK and PPS prior to injection were 371-382°C and 293-299°C, respectively. The mold temperature, injection pressure and molten temperatures of the LCP300 materials were 240°C, 110 MPa, and 450°C, respectively. For the LCP500 grade material, the mold temperature was 220°C, the injection pressure was 7 MPa and the molten temperature was 430°C. All the test specimens used for this work were 0.32 cm thick.

EXPERIMENTAL PROCEDURE

ELECTRICAL PROPERTIES

EMI shielding effectiveness values were obtained using the dual-chamber method described in ASTM E7-83. Shielding effectiveness measurements were made at 14 different frequencies ranging from 15MHz to 1GHz. Volume resistivity measurements were performed in accordance with ASTM D257-78 thru the thickness of the sample. The specimens were approximately 2.08 cm in diameter. A digital ohmmeter was used to measure the resistance of the specimens which were sandwiched between two copper plates at a pressure of 0.62 MPa.

MICROSCOPY

Scanning electron microscopy (SEM) was used to assess the fiber-matrix interfacial bonding at the tensile fracture surfaces. Optical microscopy was used to examine the void content of the materials and the distribution of the conductive reinforcements in the matrices. Various samples from the plaques and tensile specimens were mounted, polished, and examined at 100x and 800x.

MECHANICAL PROPERTIES

The tensile properties of the materials were measured according to ASTM D638-82 and the tensile strength, modulus, and strain to failure were recorded. The tensile specimens were tested as received from an end-gated injection mold. Tests were performed at room temperature and 104°C.

End-gated specimens were also tested as received to determine the flexural strength of the materials. The testing was performed at room temperature and 104°C according to ASTM D790-81 and flexural strengths were recorded.

Drop weight impact tests were conducted in accordance with ASTM D3029-78. Impact energies capable of through-penetration were exerted on 12.7 cm square specimens and the absorbed energy was determined from the force-displacement curve at peak load. With the exception of the unfilled PEEK material, all of the specimens were impacted at a velocity of approximately 168 cm/s. Failure of the unfilled PEEK required increasing the impact velocity to greater than 579 cm/s.

WATER ABSORPTION AND DENSITY

Water absorption tests were performed in accordance with ASTM D570-81. The test specimens were 7.62 cm long by 2.54 cm wide. The tests were conducted at room temperature with the samples completely immersed in distilled water.

Density measurements were made in accordance with ASTM D792-66 (reapproved 1975). The specimens were approximately 5.08 cm long by 5.08 cm wide and immersed in distilled water at room temperature.

THERMAL ANALYSIS

Differential scanning calorimetry (DSC) was used to measure heat flow (mW) as a function of temperature. Glass transition temperature (T_g), onset melting temperature (T_{mo}), peak melting temperature (T_{mp}), latent heat fusion (ΔH_f) and specific heat (C_p) values were determined using this data. T_g 's, T_{mo} 's and T_{mp} 's were calculated using the method outlined in ASTM-D-3418 (see figure 1).

The ΔH_f 's were calculated from the area of the peak. The degrees of crystallinity were estimated from the ratios of the ΔH_f 's for the polymer specimens to the ΔH_f 's for the respective 100% crystalline polymers. The ΔH_f for 100% crystalline PPS is 100 J/g [Phillips Petroleum Co.] and for 100% crystalline PEEK is 130 J/g [Imperial Chemical Industries (ICI) Plastics Division].

The C_p 's were calculated from the displacement (d) between the baseline in the absence of a sample and the sample trace. From basic thermal analysis theory, C_p of a specimen may be calculated using the following relationship [5]:

$$\left(\frac{d_s}{C_{p_s} m_s} \right) = \left(\frac{d_r}{C_{p_r} m_r} \right) \quad (2)$$

where

m = mass
s = sample
r = reference

The reference used for C_p calculations in this report was Indium.

The DSC specimens were 0.16 cm wide by 0.32 cm long. These tests were conducted under ambient atmospheric pressure with a nitrogen purge set at approximately 100 cc/min. The temperature, which was initially at 25°C, was increased at a rate of 10°C/min up to 500°C during these tests.

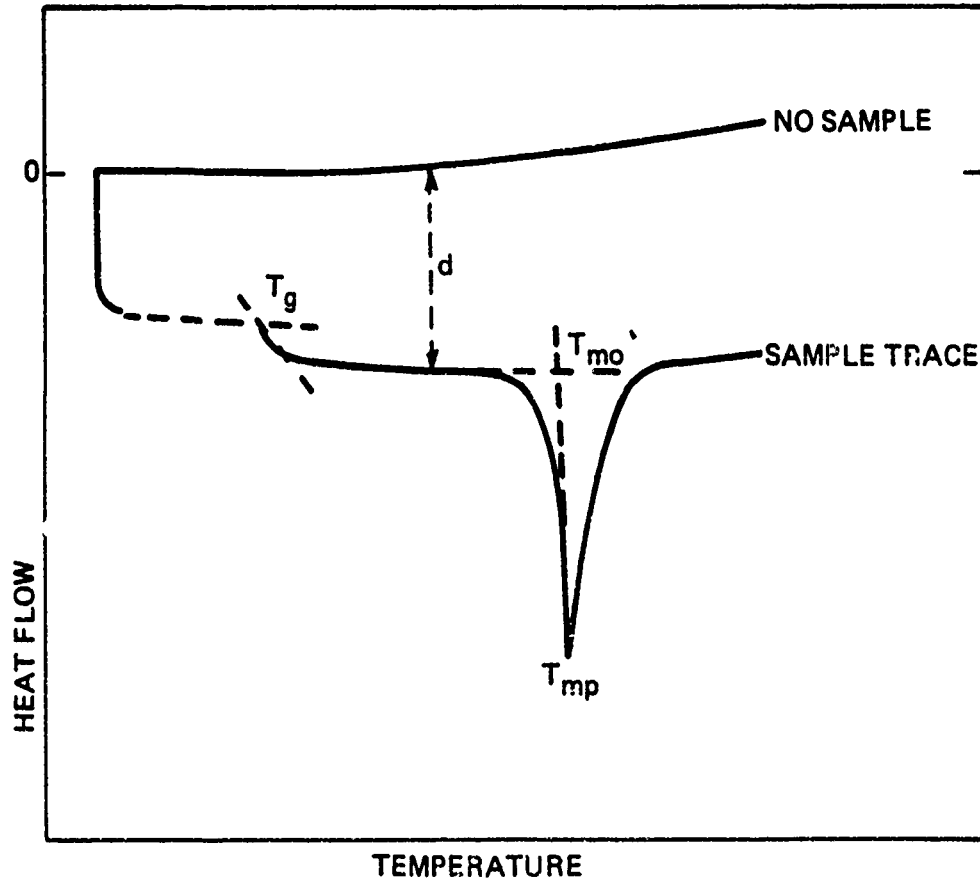


Figure 1. Sample DSC Curve

RESULTS AND DISCUSSION

ELECTRICAL PROPERTIES

EMI shielding effectiveness (SE) values are listed in table 1 in descending order of attenuation. Four of the hybridized composites (PPS/30%NiGr/15%SS, PPS/30%NiGr/10%SS, PPS/30%NiGr/20%NiFlk and PPS/25%NiGr/15%SS) provided at least 80 dB of shielding at the frequencies of 80, 100, 150, 200 and 300 MHz, respectively. Outside of these frequencies the dynamic range of the test equipment was limited (see table 1). The PPS/30%NiGr/15%SS material possessed an attenuation level greater than or equal to the dynamic range of the test equipment except at the frequencies of 150, 200, 300, and 600 MHz. However, even at these four frequencies the dynamic ranges were within the standard deviations of the PPS/30%NiGr/15%SS values. The LCP300/30%Gr composite provided the least amount of attenuation.

Several conductive composites formulated utilizing the hybrid reinforcement approach shielded more effectively than composites with a single reinforcement. The NiGr/SS combinations in PPS and PEEK provided better attenuation properties than the respective resins reinforced with 40% NiGr. This may be attributed to the ductility of the SS fibers. The SS fibers are more apt to deform while NiGr fibers are apt more to fracture during processing. Therefore, the SS fibers retain higher aspect ratios than NiGr fibers after molding. Additionally, the SS fibers are longer (0.762 cm) than NiGr fibers (0.635 cm) when supplied to the compounder. These initially longer SS fibers also lead to higher aspect ratios in the molded product. Higher aspect ratios provide a more continuous conductive network throughout the polymeric matrix which results in better electrical properties.

Loadings of 30% NiGr/20%NiFlk, 25%NiGr/10%SS, and 40% NiGr were molded into both PPS and PEEK thermoplastics respectively. The PPS composites shielded better than the PEEK materials with the same weight percent loadings. One explanation for this behavior is that PEEK composites have higher melt flow viscosities than PPS materials. The more viscous PEEK materials consequently have higher shear stresses which cause fibers to fracture. This would result in lower reinforcement aspect ratios in PEEK composites than in PPS composites. Higher reinforcement aspect ratios in a composite should provide an efficient conductive network throughout the material which would enable the composite to effectively absorb and reflect incident energy signals. Also, PPS composites had approximately one percent by volume more reinforcements than comparable PEEK materials. Another possible explanation may be the lower degree of crystallinity observed in PEEK than in PPS (see thermal analysis). The spherulitic growth in more highly crystalline PPS may cause the fibers and flakes to be located in the amorphous regions between the growing nuclei. The smaller amorphous regions in PPS composites would induce better electrical contacts between the conductive reinforcements than in the larger amorphous regions in PEEK composites. Good contacts between the reinforcements should also enhance the EMI shielding effectiveness of the composite. It was not determined

TABLE 1. EMI SHIELDING EFFECTIVENESS (dB ATTENUATION)

MATERIAL SYSTEM		FREQUENCY (MHz)											SPECIMEN THICKNESS (in)				
		15	20	30	40	60	80	100	150	200	300	400			600	800	1000
Dynamic Range Al 2024	AV 64	66	**	**	**	**	**	**	**	92	95	92	78	62	67	72	0.120
	SD 0	1	1	1	1	0	0	0	0	0	0	0	0	1	0	1	
PPS/30NiGr/15SS	AV **	**	**	**	**	**	**	**	**	90+	87+	90+	**	60+	**	**	0.125
	SD **	**	**	**	**	**	**	**	**	2+	9+	2+	**	3+	**	**	
PPS/30NiGr/10SS	AV **	**	**	**	**	**	**	**	**	91+	90	88	70+	**	**	**	0.123
	SD **	**	**	**	**	**	**	**	**	1+	2	1	13+	**	**	**	
PPS/30NiGr/20NiFlk	AV **	**	**	**	**	**	**	80	89	90	87	77	**	**	**	69+	0.124
	SD **	**	**	**	**	**	**	2	2	3	2	1	**	**	**	5+	
PPS/25NiGr/15SS	AV **	**	**	**	**	**	**	**	88	84	88	**	**	**	**	**	0.127
	SD **	**	**	**	**	**	**	**	2	7	1	**	**	**	**	**	
PPS/25NiGr/10SS	AV **	**	**	**	**	74+	73	72	69	74	84	77	**	**	**	**	0.128
	SD **	**	**	**	**	1+	1	2	2	2	2	1	**	**	**	**	
PPS/40NiGr	AV **	**	**	**	**	73	74	73	75	72	83	74	60+	**	**	**	0.122
	SD **	**	**	**	**	1	3	2	5	3	6	1	2+	**	**	**	
PPS/40NiGr/15NiFlk	AV **	**	68	67	63	63	63	63	63	63	71	76+	**	**	65	69	0.121
	SD **	**	0	1	1	1	1	1	1	1	2	2+	**	**	1	1	
PPS/25NiGr/10Gr	AV **	65	68	66	62	63	63	62	61	61	72	68	61	61	60	64	0.123
	SD **	1	1	2	1	1	1	1	1	1	4	3	1	2	1	1	
PPS/20NiGr/30Gr	AV **	65	65	63	59	59	59	58	55	56	63	68	55	55	50	55	0.123
	SD **	1	2	1	0	1	1	1	1	1	1	6	1	1	1	1	
PEEK/25NiGr/10SS	AV 62	63	63	68	66	66	58	60	56	53	60	57	61+	63	63	70	0.128
	SD 1	1	3	3	9	9	1	4	1	2	1	1	2+	1	1	1	

** Meets or exceeds the dynamic range of test equipment.

+ Indicates at least one of the readings meets or exceeds the dynamic range.

TABLE 1. EMI SHIELDING EFFECTIVENESS (dB ATTENUATION) (Continued)

MATERIAL SYSTEM	FREQUENCY (MHz)														SPECIMEN THICKNESS (in)
	15	20	30	40	60	80	100	150	200	300	400	600	800	1000	
PPS/20Gr/15SS	AV 62	63	63	62	66	62	56	56	57	60	46	43	38	42	0.123
	SD 1	1	1	1	9	5	1	1	1	1	3	2	1	1	
PPS/40Gr	AV 62	62	62	58	55	54	52	49	48	56	63	47	39	43	0.123
	SD 1	1	1	1	1	1	1	1	0	1	2	1	1	1	
PPS/10NiGr/25Gr	AV 61	60	58	54	62	49	47	44	42	51	57	39	30	33	0.123
	SD 1	1	1	1	10	1	1	1	1	2	6	1	1	1	
PEEK/40NiGr	AV 51	49	46	44	40	38	39	40	42	58	56	46	38	41	0.120
	SD 1	1	1	1	1	1	1	1	2	4	2	1	1	1	
PEEK/30NiGr/ 20NiFlk	AV 52	49	47	44	41	40	39	40	44	55	49	41	32	36	0.121
	SD 1	0	0	1	1	1	1	1	1	2	3	0	0	1	
LCP300/30Gr	AV 45	43	41	37	33	33	34	35	35	42	30	23	14	17	0.122
	SD 1	1	1	1	1	1	1	2	5	2	3	1	1	1	

** Meets or exceeds the dynamic range of test equipment.

+ Indicates at least one of the readings meets or exceeds the dynamic range.

if the reinforcements had higher aspect ratios or if there were better contacts between the reinforcements in PPS than in PEEK. Accurate fiber length measurements are difficult to obtain from photomicrographs because the fibers are usually slightly mis-oriented into the plane of the picture and hence they appear shorter than they actually are. Also, quantitative assessment of contacts between reinforcements was not determined for the same reason.

Volume resistivity results are listed in table 2 in descending order. Composite resistivities ranged from 0.6 ohm-cm for PPS/30%NiGr/10%SS to 3000 ohm-cm for LCP300/30%Gr. Combinations of 25 to 30% by weight NiGr with 10 to 15% by weight SS yielded low resistivity values. The LCP300/30%Gr material yielded a mean resistivity value an order of magnitude higher than that reported for PPS/30%Gr [2]. However, this may be due to poor fiber dispersion in the LCP300 matrix. The large standard deviation on the LCP300/30%Gr resistivity supports the theory that resin and fiber rich areas exist within the matrix. The cause of non-uniformities in the LCP300 matrix was not investigated. The resistivities reported for PEEK/25%NiGr/10%SS and PEEK/40%NiGr were within the standard deviation of the resistivities for the PPS resin filled with the same weight loadings, respectively. However, the resistivity of PEEK/30%NiGr/20%NiFlk was much higher than the PPS/30%NiGr/20%NiFlk value. The possible reasons given to explain the better shielding values of the PPS composites compared to the PEEK composites at similar loadings may also be applied to explain the resistivity results.

The PPS/30%NiGr/10%SS had similar shielding and resistivity values to PPS/30%NiGr/15%SS. Hence, the additional 5%SS in the latter composite did not significantly improve the electrical properties. A possible reason for these similar properties is that the upper limits on EMI attenuation and electrical conductivity are close to being attained with PPS/30%NiGr/10%SS. However, PPS/25%NiGr/15%SS had higher EMI protection and lower resistivity values than PPS/25%NiGr/10%SS. These two composites both have lower volume percent reinforcements (see table 5) than PPS filled with 30%NiGr/10%SS and 30%NiGr/15%SS, respectively. Therefore, it may be possible to improve the electrical properties of PPS/25%NiGr/15%SS by formulating this type of hybrid with higher SS fiber concentrations.

The Gr fiber reinforced PPS composites did not perform as well electrically as the NiGr fibers or the hybrid combinations.

MICROSCOPY

Optical microscopy was used to examine the microstructural properties of the conductive composites. The microscopy work revealed that all of the materials had voids. The PPS composites that were loaded with the NiGr/SS fiber combinations appeared to contain the largest voids and the highest void fractions. The voids varied in size and number from material to material but in general the PEEK systems appeared to have lower void contents than the PPS systems.

TABLE 2. VOLUME RESISTIVITY RESULTS

MATERIAL SYSTEM		VOLUME RESISTIVITY (OHM-CM)
LCP300/30Gr	AVE	3000
	SD	2270
PPS/10NiGr/25Gr	AVE	30.9
	SD	2.0
PPS/20Gr/15SS	AVE	22.8
	SD	0.9
PPS/40NiGr	AVE	22.2
	SD	15.0
PEEK/30NiGr/20NiFlk	AVE	15.2
	SD	4.3
PEEK/40NiGr	AVE	13.7
	SD	13.0
PPS/40Gr	AVE	9.71
	SD	1.03
PPS/25NiGr/10Gr	AVE	7.40
	SD	1.03
PPS/20NiGr/30Gr	AVE	6.83
	SD	0.28
PPS/40NiGr/15NiFlk	AVE	3.91
	SD	1.30
PPS/25NiGr/10SS	AVE	2.80
	SD	0.58
PEEK/25NiGr/10SS	AVE	2.63
	SD	0.71
PPS/30NiGr/20NiFlk	AVE	1.64
	SD	0.12
PPS/25NiGr/15SS	AVE	0.97
	SD	0.09
PPS/30NiGr/15SS	AVE	0.61
	SD	0.07
PPS/30NiGr/10SS	AVE	0.60
	SD	0.06

Microscopy also showed that the nickel plating separated from the graphite fibers in the NiGr fiber systems. PPS/40%NiGr and PEEK/40%NiGr materials are shown in figures 2 and 3 at both 100x and 800x magnification. Some separation of the nickel plating from the graphite fibers can be observed in the high magnification photograph. Since only the molded material was examined, it could not be determined whether the debonding occurred before compounding, during compounding or during the molding process.

Most of the materials exhibited good dispersion of the reinforcements throughout the thermoplastic matrix, however some agglomeration was present. The materials that were filled with SS and NiGr fibers occasionally contained small areas of SS and NiGr clumping. The microstructure of PPS/25%NiGr/10%SS is shown in figure 4. The materials filled with NiGr/NiFlk combinations also displayed some agglomeration of the reinforcements, however to a lesser degree than the NiGr/SS systems. The dispersion characteristics of the PPS/40%NiGr/15%NiFlk hybrid composite is shown in figure 5. Examples of these agglomerations are presented in figure 6 at 900x magnification.

SEM was used to examine the fracture surfaces of the tensile specimens. All of the systems that contained NiGr fibers showed poor fiber adhesion to the matrix. The fiber pull-out that resulted can be observed in the figure 7. It can be seen from this figure that the Ni plating has separated from some of the graphite fibers. Since this debonding of the nickel plating was also observed in the optical micrographs of the unstressed samples, it could not be determined whether the fracture mechanism was primarily due to the failure of the nickel/matrix interfacial bonding, the failure of the graphite/nickel interfacial bonding, or a combination of both. The NiFlk materials also exhibited poor bonding to the thermoplastics. A small gap between the nickel flake and the PPS matrix shown in figure 8 depicts the poor adhesion qualities of this filler. The SS fibers also exhibited poor adhesion and had little residual resin left on the fibers after fracture. The SS fibers in figure 9 can be distinguished from the NiGr fibers by their deformation after failure. Two of the deformed SS fibers can be seen in the lower portion of the photograph. Poor adhesion of the graphite fibers to the LCP300 resin can be observed in figure 10. Fiber pull-out is evident, and little residual resin remained on the exposed fibers. The fibril microstructural characteristics of the LCP resin appears to be quite different from the smooth brittle fracture surface of the PPS. Figure 11 shows the fracture surface of the PEEK thermoplastic that was loaded with 40% NiGr. The failure mechanism here is fiber pull-out which is very similar to that shown in figure 7 for the PPS/40%NiGr composite. The fracture surface of the PPS material appears to be smoother than the PEEK resin. This phenomenon may be attributed to the inherently brittle nature of PPS.

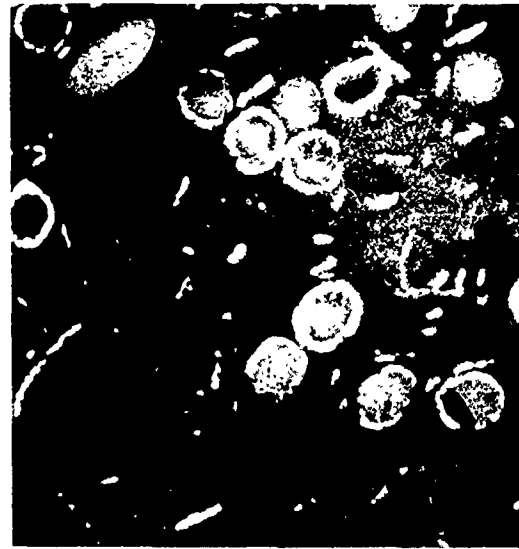


Figure 2. Photomicrographs (100x and 800x) of PPS/40%NiGr

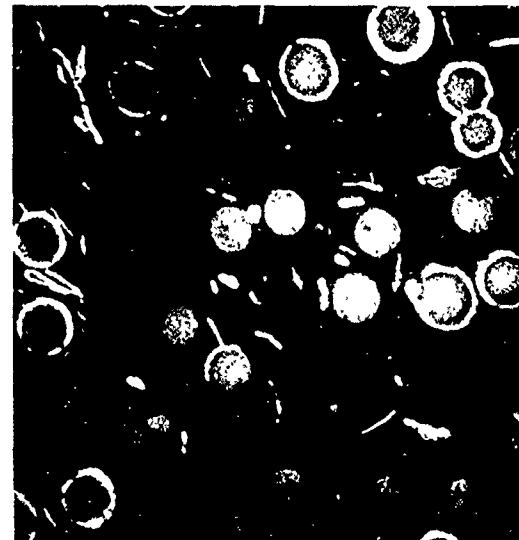
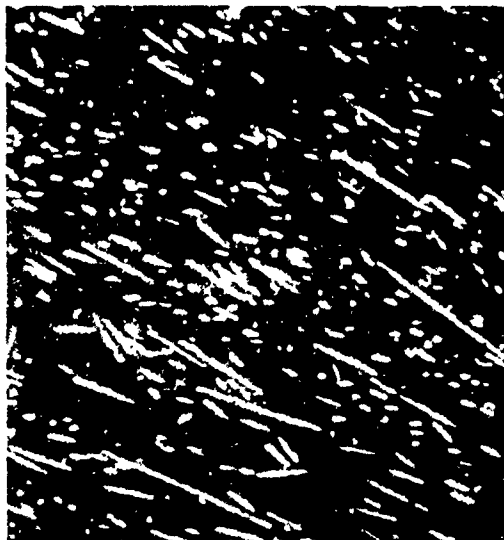


Figure 3. Photomicrographs (100x and 800x) of PEEK/40%NiGr

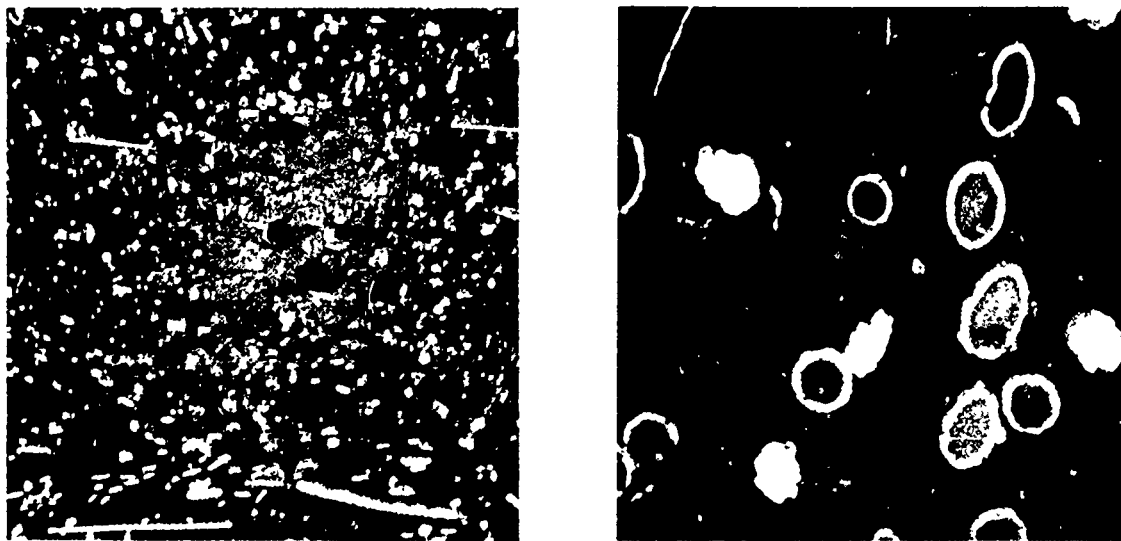


Figure 4. Photomicrographs (100x and 800x) of PPS/25%NiGr/10%SS

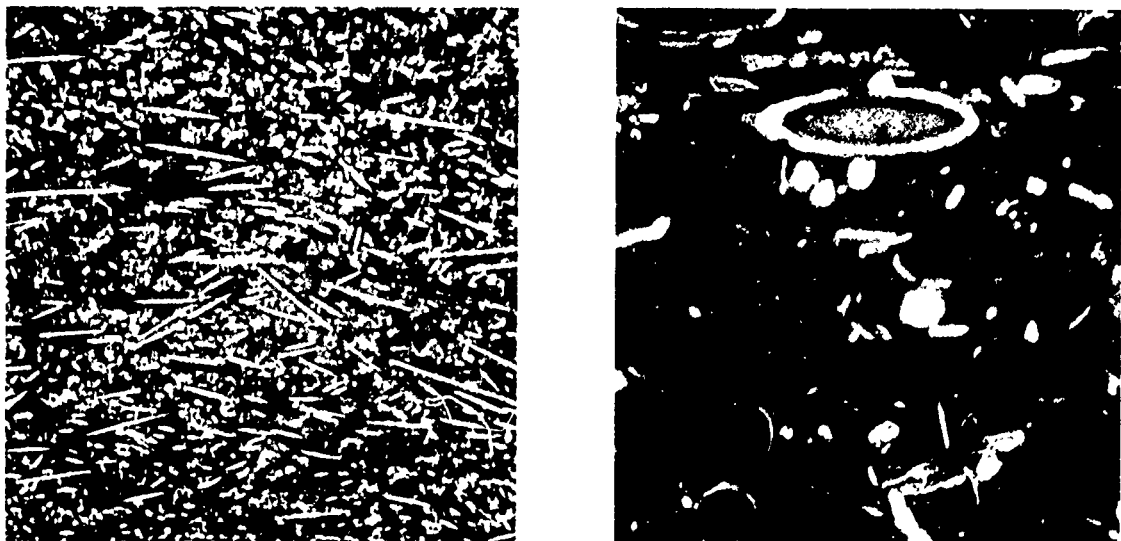
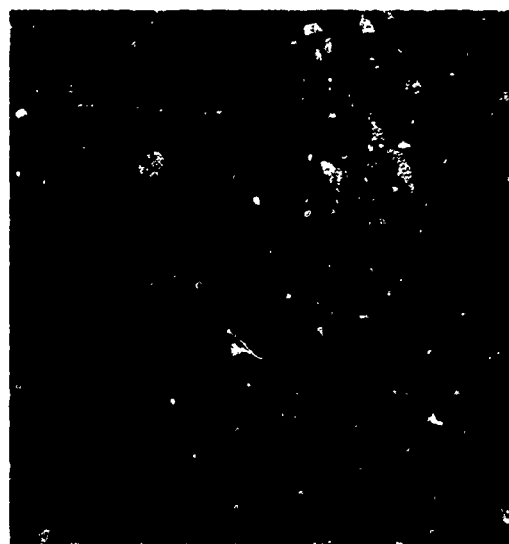


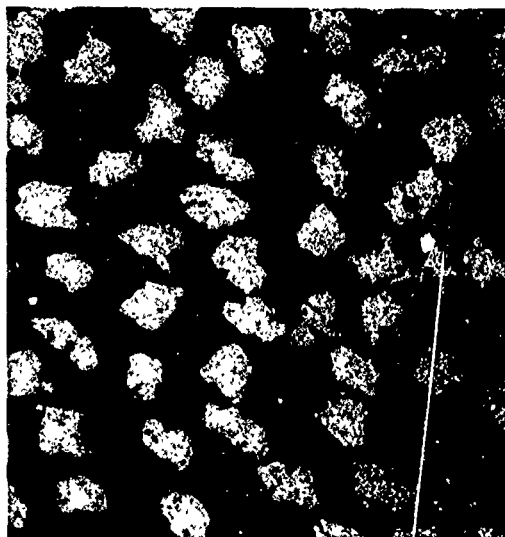
Figure 5. Photomicrographs (100x and 800x) of PPS/40%NiGr/15%NiFlk



(a)



(b)



(c)

Figure 6. Photomicrographs (900x) of Reinforcement Agglomeration (a) NiGr Fibers, (b) NiFlk, and (c) SS Fibers



Figure 7. SEM Photograph (2000x) of
PPS/40%NiGr Tensile
Fracture Surface



Figure 8. SEM Photograph (2000x) of
PPS/30%NiGr/20%NiFlk
Tensile Fracture Surface



Figure 9. SEM Photograph (500x)
PPS/30%NiGr/10%SS Tensile
Fracture Surface

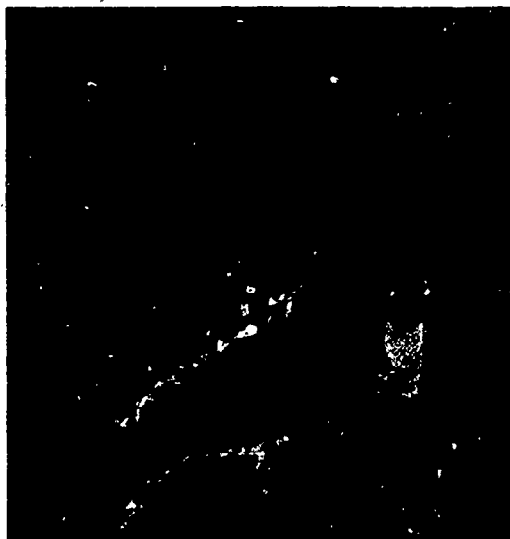


Figure 10. SEM Photograph (1000x) of LCP300/30%Gr
Tensile Fracture Surface



Figure 11. SEM Photograph (2000x) PEEK/40%NiGr
Tensile Fracture Surface

MECHANICAL PROPERTIES

The tensile properties of the various material systems are shown in table 3 in descending order of room temperature tensile strength. Failure stress, Young's modulus and strain to failure at room temperature and at 104°C are listed. Cast 355 aluminum and 7075-T6 aluminum values are shown for comparative purposes.

Of the unfilled materials examined, the LCP's displayed the highest strength and stiffness values. The unfilled PEEK resin exhibited ductile failure at room temperature and at 104°C. This is reflected in its strain-to-failure values which exceeded the range of the extensometer (2.5%) used during testing. The unfilled PPS polymer also exhibited ductile failure when tested at 104°C, but failed in a brittle manner at room temperature. This effect was expected since the glass transition temperature of PPS was 88.8°C. The unfilled PEEK had a comparable modulus to that of PPS at both temperatures, however PEEK had higher strength values.

The addition of conductive reinforcements to the above matrices generally enhanced the tensile strength and modulus of the unfilled polymers, but had a deleterious effect on the strain-to-failure. An exception to this trend was the unfilled LCP300 material which displayed a higher tensile strength than the LCP300/30%Gr. Poor fiber/matrix adhesion may have contributed to this. Furthermore, the addition of fibers into the system during molding may have disrupted the LCP's chain alignment. This effect would have degraded the self-reinforcing characteristics found in this polymer.

Both PPS and PEEK composites exhibited their highest strength values when filled with 40% by weight NiGr fibers. The PPS/NiGr/NiFlk combinations displayed intermediate strengths and the PPS/NiGr/SS hybrids were the weakest of these combinations. Furthermore, increasing the SS content in PPS from 10% to 15% in combination with 25% or 30%NiGr resulted in lower tensile strengths. The PPS/NiGr/SS combinations may have been weaker than the PPS/40%NiGr for several reasons. First, poor SS fiber/PPS adhesion (see figure 9) may have caused these lower strength properties. Also, microscopy showed that the PPS/NiGr/SS hybrids tended to contain the largest voids. It has been shown that void content has a deleterious effect on the composite strength properties [6]. PPS/40%NiGr contained a higher volume percentage of reinforcements than most of the PPS/NiGr/SS systems (see table 6) and therefore higher strength was expected.

The PPS/40%Gr exhibited both the highest volume fraction of reinforcements and the highest elastic modulus out of all the thermoplastic-based materials tested. The NiGr/SS reinforcement schemes provided the least amount of stiffness compared to the other composites. Additionally, none of the filled systems failed in the ductile manner that was exhibited by the PPS and PEEK neat resins at 104°C.

TABLE 3. TENSILE PROPERTIES

MATERIAL SYSTEM	----- ROOM TEMP -----			----- 104°C -----		
	STRESS (MPa)	MODULUS (GPa)	STRAIN (%)	STRESS (MPa)	MODULUS (GPa)	STRAIN (%)
Al 7075	* 496.4	71.0	0.90	434.4	-----	-----
Al 355 Cast	* 165.5	71.0	0.43	158.6	-----	-----
PEEK/40NiGr	AV 156.5	15.2	1.12	113.8	15.9	1.01
	SD 6.2	1.7	0.15	9.7	1.0	0.05
PEEK/30NiGr/ 20NiFlK	AV 135.1	16.8	0.88	104.1	16.8	0.81
	SD 10.3	1.0	0.05	5.5	3.0	0.14
PEEK/25NiGr/ 10SS	AV 135.1	13.9	1.22	92.4	13.6	1.07
	SD 4.8	1.2	0.11	4.1	2.0	0.13
PPS/40NiGr	AV 133.1	19.1	0.64	75.2	**	1.19
	SD 10.3	4.0	0.13	2.1	**	0.16
PPS/30NiGr/ 20NiFlK	AV 132.4	16.3	0.87	68.9	11.0	1.11
	SD 8.3	1.4	0.10	3.4	1.9	0.02
LCP500/Unfilled	AV 131.7	20.5	1.17	68.3	14.5	0.67
	SD 4.8	2.4	0.12	8.3	0.9	0.16
PPS/40Gr	AV 125.5	24.1	0.62	84.9	**	1.17
	SD 12.4	3.2	0.07	7.6	**	0.27
PPS/40NiGr/ 15NiFlK	AV 125.5	20.1	0.98	71.7	11.9	1.15
	SD 15.2	1.9	0.31	4.1	5.9	0.14
LCP300/Unfilled	AV 117.2	14.6	1.10	78.6	-----	-----
	SD 14.5	2.5	0.20	7.6	-----	-----
PEEK/Unfilled	AV 84.8	3.3	***	53.8	3.0	***
	SD 2.1	0.2	***	0.7	0.1	***
PPS/25NiGr/10SS	AV 77.9	10.8	0.76	43.4	7.1	1.47
	SD 2.8	1.7	0.12	2.8	1.0	0.18
PPS/30NiGr/10SS	AV 69.6	11.9	0.61	38.6	8.5	1.09
	SD 8.3	6.6	0.04	2.8	1.0	0.15
LCP300/30Gr	AV 68.9	17.7	0.51	49.6	12.6	0.72
	SD 20.7	1.9	0.25	4.8	1.5	0.06
PPS/25NiGr/15SS	AV 64.8	10.2	0.72	37.9	6.3	1.51
	SD 4.8	1.0	0.16	1.4	0.7	0.09
PPS/30NiGr/15SS	AV 60.7	11.9	0.51	38.6	7.4	1.26
	SD 4.1	1.0	0.05	3.4	1.2	0.14
PPS/Unfilled	AV 26.2	3.3	0.78	22.8	3.0	***
	SD 2.1	0.2	0.08	1.4	0.4	***

* Battelle Columbus Laboratories.

** No linear portion of stress strain curve.

*** Elongation exceeds the range of strain measurement equipment.

In general, the PEEK systems performed better mechanically than the PPS systems. This effect was expected since the unfilled PEEK has approximately four times the room temperature strength as the unfilled PPS. Furthermore, since PEEK has a higher glass transition temperature than PPS (as shown in table 9) it is expected that the PEEK materials have a lower percent reduction in tensile strength at elevated temperatures as compared to the PPS materials. The results in table 3 verify this experimentally.

The materials' flexural properties are shown in table 4 in descending order of room temperature flexural strength. The unfilled PEEK and LCP matrices had flexural strengths at least two times that of the unfilled PPS resin. At room temperature, the LCP300 material showed a slightly higher flexural strength and more than four times the stiffness than that of the neat PEEK thermoplastic. However, at 104°C the LCP300 resin exhibited a slightly lower failure stress and more than three times the stiffness than that of PEEK. The high stiffness of the LCP300 material may be attributed to the self reinforcing polymer chain alignment that is characteristic of this liquid crystal polymer.

Of the filled systems, the PEEK composites that contained SS fibers generally had superior flexural strengths. This may be attributed to low void contents and better fiber interfacial bonding characteristics as compared to the PPS composites. The PPS/30%NiGr/15%SS system showed about 18% lower flexural strength than the PPS/40%NiGr system although both materials contained similar fiber volume fractions. The three reinforced PEEK composites were in the top five materials listed in table 4. The PPS composites generally displayed the highest stiffness values at room temperature, but the PEEK composites had the highest flexural strengths at 104°C. The LCP systems displayed low flexural strengths at both test temperatures. The addition of graphite to LCP300 had little effect on the flexural strength, but it did result in a 30% increase in modulus at room temperature.

Impact energies required to initiate failures are presented in table 5 in descending order of impact strength. Of the unfilled thermoplastics evaluated, PEEK demonstrated the highest resistance to impact. The PEEK material absorbed more than 120 joules. The unfilled LCP500 and LCP300 absorbed 5.79 joules and 4.39 joules, respectively, while the unfilled PPS absorbed only 0.18 joules.

Once the polymers were filled with the conductive reinforcements, their impact properties changed substantially. The impact properties of the PEEK were degraded, the impact strength of the LCP300 increased slightly and the impact strength of the PPS dramatically increased with the addition of reinforcements. In general, the filled PEEK systems performed better than the filled PPS and LCP systems.

TABLE 4. FLEXURAL PROPERTIES

MATERIAL SYSTEM	-- ROOM TEMP --		---- 104°C ----	
	STRESS (MPa)	MODULUS (GPa)	STRESS (MPa)	MODULUS (GPa)
PEEK/25NiGr/10SS	AV 238.6	13.86	168.2	11.45
	SD 5.5	0.48	3.4	0.41
PPS/30NiGr/20NiFlk	AV 229.6	19.17	116.5	9.72
	SD 5.5	1.10	8.3	0.83
PEEK/40NiGr	AV 226.1	14.82	199.9	13.03
	SD 7.6	0.34	4.8	0.41
PPS/40NiGr	AV 217.2	23.72	137.9	12.82
	SD 5.5	1.38	9.0	2.41
PEEK/30NiGr/20NiFlk	AV 215.8	15.31	183.4	11.58
	SD 11.0	0.48	10.3	1.38
PPS/40Gr	AV 191.0	17.72	160.6	16.34
	SD 8.3	1.86	4.8	2.14
PPS/40NiGr/15NiFlk	AV 180.6	19.58	107.6	8.20
	SD 20.7	1.72	4.1	0.34
PPS/25NiGr/10SS	AV 158.6	14.34	84.1	5.65
	SD 11.7	0.83	2.8	0.41
PPS/30NiGr/15SS	AV 155.1	16.75	75.8	7.86
	SD 17.2	0.69	5.5	0.76
PPS/30NiGr/10SS	AV 153.1	16.34	74.5	6.27
	SD 12.4	0.69	6.2	0.48
LCP300/Unfilled	AV 152.4	10.48	84.8	7.03
	SD 20.7	0.83	2.8	1.03
LCP300/30Gr	AV 145.5	13.31	86.9	7.17
	SD 15.2	0.55	2.1	0.76
PEEK/Unfilled	AV 143.4	2.55	91.0	2.14
	SD 4.8	0.21	2.8	0.14
PPS/25NiGr/15SS	AV 137.9	15.38	77.9	5.38
	SD 8.3	0.90	4.8	0.55
LCP500/Unfilled	AV 128.2	12.20	83.4	7.93
	SD 13.1	2.62	2.8	0.41
PPS/Unfilled	AV 55.2	3.17	47.0	0.48
	SD 5.6	0.48	5.7	0.07

TABLE 5. IMPACT PROPERTIES

MATERIAL SYSTEM		ENERGY ABSORBED (J)
PEEK/Unfilled	AVE	>120
	SD	-----
PEEK/25NiGr/10SS	AVE	9.10
	SD	1.61
PEEK/40NiGr	AVE	8.69
	SD	0.91
PPS/30NiGr/20NiFlk	AVE	8.08
	SD	2.39
PEEK/30NiGr/20NiFlk	AVE	7.31
	SD	1.10
LCP300/30Gr	AVE	6.97
	SD	0.89
LCP500/Unfilled	AVE	5.79
	SD	1.86
PPS/30NiGr/15SS	AVE	4.70
	SD	0.39
PPS/25NiGr/15SS	AVE	4.41
	SD	0.26
LCP300/Unfilled	AVE	4.39
	SD	0.72
PPS/25NiGr/10SS	AVE	4.28
	SD	0.39
PPS/40NiGr/15NiFlk	AVE	3.92
	SD	0.99
PPS/40NiGr	AVE	3.89
	SD	0.38
PPS/30NiGr/10SS	AVE	3.80
	SD	0.15
PPS/40Gr	AVE	3.67
	SD	0.50
PPS/Unfilled	AVE	0.18
	SD	0.04

In summary, the PEEK composites showed better strength and impact properties than PPS composites. Those systems that contained NiGr/SS combinations generally showed the poorest mechanical properties. This may be attributed to poor fiber interfacial bonding characteristics and high void contents. The NiGr/NiFlk hybrids and materials filled with NiGr fibers generally performed better than the NiGr/SS composites.

PHYSICAL PROPERTIES

Calculated and measured densities appear in table 6 in descending order of measured density values. All of the composites have measured densities less than 71 percent of that of aluminum. The composites with the highest densities contained NiGr/NiFlk combinations followed by the NiGr/SS fiber systems. Calculated density values were determined using the measured neat resin densities and the densities of the conductive fibers and flakes reported by the manufacturers. The measured densities of LCP500, PPS, LCP300, and PEEK were 1.38, 1.37, 1.35, and 1.29 g/cc, respectively, and the reported densities of NiGr, NiFlk, Gr, and SS are 3.2, 7.93, 1.8 and 7.9 g/cc, respectively. Comparisons between calculated and measured values are shown in the percent difference column of table 6. Most of the composites had less than a 5% difference between the calculated and measured density values. Only PPS/40%NiGr/15%NiFlk had a difference greater than 7.5%. These percent difference values may be attributed to voids in the composites. These values support the microscopy observations that the PPS composites have higher void contents than PEEK composites with similar loadings. Also listed in table 6 are calculated volume percentages of reinforcements for each of the composites. Volume percentages were calculated using appropriate resin and reinforcement density data. Values ranged from 15.2 percent for PEEK/25%NiGr/10%SS to 38.6 percent for PPS/20%NiGr/30%Gr.

Water absorption results are listed in table 7 in descending order of percent weight gain at saturation. All of the materials absorbed less than one percent by weight of water except for PPS/40%NiGr/15%NiFlk and PPS/30%NiGr/10%SS. Of the unfilled polymers, PEEK absorbed the most water after 24 hours and at saturation, followed by PPS and LCP300, respectively. However, PPS composites absorbed the most water with saturation values ranging from 0.285 percent for PPS/10%NiGr/25%Gr to 1.404 percent for PPS/40%NiGr/15%NiFlk. PEEK composite results ranged from 0.183 percent for PEEK/30%NiGr/20%NiFlk to 0.284 percent for PEEK/25%NiGr/10%SS. LCP300/30%Gr had 0.021 percent absorption at saturation which was the lowest of all the materials tested. Note that the reinforced PPS materials all absorbed more water than the unfilled matrix while the PEEK and LCP300 composites absorbed less water than their respective unfilled resins. The behavior of the PPS composites may be attributed to the higher void contents in the PPS matrices compared to the PEEK and LCP matrices. Examples of higher void contents in PPS composites may be observed in figures 2, 4 and 5. The PPS composites that absorbed the most water had the highest void contents as indicated by the larger percent differences between calculated and measured densities

TABLE 6. DENSITY VALUES

MATERIAL SYSTEM	MEASURED DENSITY (G/CC)	CALCULATED DENSITY (G/CC)	PERCENT DIFFERENCE	CALCULATED VOLUME % REINFORCEMENTS
A1 7075	AVE 2.79 SD 0.00	2.80	0.2	
PPS/30NiGr/ 20NiFlk	AVE 1.98 SD 0.00	2.07	4.3	24.6
PPS/40NiGr/ 15NiFlk	AVE 1.90 SD 0.00	2.12	10.4	30.5
PEEK/30NiGr/ 20NiFlk	AVE 1.90 SD 0.00	1.97	3.6	23.5
PPS/30NiGr/15SS	AVE 1.85 SD 0.01	1.94	4.6	21.9
PPS/25NiGr/15SS	AVE 1.78 SD 0.01	1.87	4.8	18.1
PPS/30NiGr/10SS	AVE 1.73 SD 0.01	1.84	6.0	19.5
PEEK/25NiGr/ 10SS	AVE 1.68 SD 0.00	1.68	0.0	15.2
PPS/25NiGr/10SS	AVE 1.67 SD 0.00	1.77	5.6	16.0
PPS/40NiGr	AVE 1.65 SD 0.01	1.78	7.3	22.2
PPS/20NiGr/30Gr	AVE 1.61 SD 0.00	1.68	4.2	38.6
PPS/20Gr/15SS	AVE 1.61 SD 0.00	1.65	2.4	21.5
PEEK/40NiGr	AVE 1.60 SD 0.00	1.69	5.3	21.2
PPS/25NiGr/10Gr	AVE 1.58 SD 0.00	1.64	3.7	21.9
PPS/10NiGr/25Gr	AVE 1.51 SD 0.00	1.55	2.6	26.3
PPS/40Gr	AVE 1.50 SD 0.01	1.51	0.7	33.7
LCP300/30Gr	AVE 1.47 SD 0.01	1.46	-0.7	24.3

TABLE 7. WATER ABSORPTION (PERCENT WEIGHT GAIN)

MATERIAL SYSTEM		24 HOURS	SATURATION	NUMBER OF DAYS
PPS/40NiGr/15NiFlk	AVE	0.065	1.404	189
	SD	0.005	0.025	
PPS/30NiGr/10SS	AVE	0.208	1.151	119
	SD	0.114	0.022	
PPS/40NiGr	AVE	0.082	0.822	119
	SD	0.014	0.055	
PPS/25NiGr/10SS	AVE	0.073	0.700	105
	SD	0.003	0.008	
PPS/30NiGr/15SS	AVE	0.073	0.677	105
	SD	0.003	0.010	
PPS/25NiGr/15SS	AVE	0.040	0.499	76
	SD	0.026	0.031	
PPS/30NiGr/20NiFlk	AVE	0.063	0.459	76
	SD	0.024	0.007	
PPS/20NiGr/30Gr	AVE	0.012	0.447	77
	SD	0.061	0.017	
PPS/40Gr	AVE	0.031	0.419	77
	SD	0.002	0.006	
PPS/20Gr/15SS	AVE	0.035	0.362	77
	SD	0.004	0.012	
PPS/25NiGr/10Gr	AVE	0.013	0.338	77
	SD	0.032	0.021	
PEEK/Unfilled	AVE	0.075	0.303	42
	SD	0.013	0.009	
PPS/10NiGr/25Gr	AVE	0.027	0.285	77
	SD	0.001	0.003	
PEEK/25NiGr/10SS	AVE	0.050	0.284	62
	SD	0.001	0.013	
PEEK/40NiGr	AVE	0.052	0.255	49
	SD	<0.001	0.007	
PEEK/30NiGr/20NiFlk	AVE	0.035	0.183	49
	SD	<0.001	0.006	
PPS/Unfilled	AVE	0.008	0.080	42
	SD	0.007	0.007	
LCP/Unfilled	AVE	0.004	0.037	35
	SD	0.004	0.009	
LCP300/30Gr	AVE	-0.004	0.021	35
	SD	0.025	0.027	

(see table 6). Note also that there appears to be fewer voids in the PPS composites which have high loadings of graphite fibers than with PPS containing large loadings of NiGr and SS fibers.

THERMAL ANALYSIS

DSC results are listed in table 8 in order of increasing peak melting temperature. Cp values for unfilled resins are shown in figure 12. From these results, note that the reinforcements did not appear to significantly affect the melting temperatures of the thermoplastics (e.g., unfilled PPS: Tmp = 279.2°C, PPS composites Tmp = 274.8 to 281.7°C).

The Tg's for unfilled PPS, PEEK, LCP300 and LCP500 are 88.8°C, 130.6°C, 117.8°C and 123.4°C, respectively. In general, the glass transition of the unfilled polymers are more easily detected because they are sharper than the glass transitions of the composites. The Tg's were not discernible for PPS composites. One possible explanation is that the polymers in the composites are more crystalline because the reinforcements offer more sites for nucleation to occur.

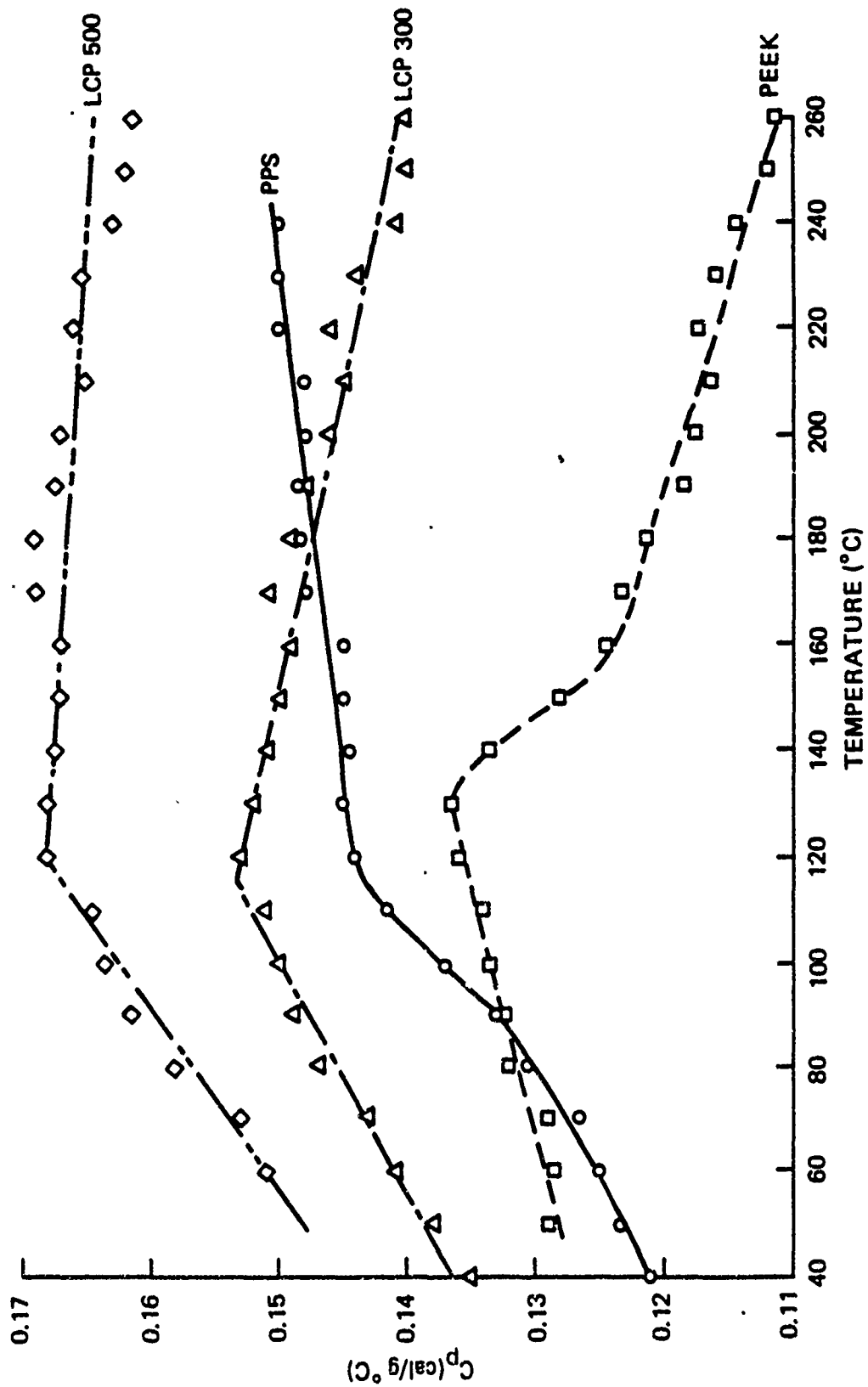
Heats of fusion for all the materials are also listed in table 8. The composites had lower ΔH_f values than their respective unfilled resins because only the matrix melted at the temperatures tested. The degree of crystallinity for unfilled PPS and unfilled PEEK from thermal analysis were 47.5% and 24.7%, respectively, as compared to values of 45.5% for PPS and 17.2% for PEEK calculated from density measurements. Amorphous and 100% crystalline density values for PPS are 1.32 g/cc and 1.43 g/cc [8], respectively and for PEEK are 1.265 g/cc and 1.41 g/cc, respectively [ICI Plastics Division]. Degrees of crystallinity for composites were not calculated due to difficulty in obtaining representative specimens. Also, crystallinity in the PPS specimens may change as they are heated [9]; although, the crystallinity of unfilled PPS determined by thermal analysis agreed well with that determined by density measurements. The crystallinity studies have shown that while the processing techniques used for PPS produced specimens close to their maximum observed crystallinity of 53% [9], the processing technique used for molding PEEK produced specimens with only half the maximum crystallinity of 48% [ICI Plastics Division]. This suggests that annealing the PEEK may be necessary to ensure stability of properties upon exposure to higher temperatures.

The ΔH_f for LCP300 and LCP500 are 3.0 J/g and 3.5 J/g, respectively. No melting peak was observed for the LCP300/30%Gr (figure 13). The ΔH_f 's for LCP300 and LCP500 are both substantially lower than for the PPS and PEEK resins.

TABLE 8. THERMAL ANALYSIS - DSC

MATERIAL SYSTEM		T _g (°C)	T _{m0} (°C)	T _{mp} (°C)	HEAT OF FUSION (J/g)
PPS/Unfilled	AVE	88.8	265.8	279.2	47.5
	SD	6.2	1.1	1.2	1.2
PPS/30NiGr/10SS	AVE	-----	256.7	274.8	44.7
	SD	-----	0.6	0.4	5.2
PPS/25NiGr/15SS	AVE	-----	259.6	275.2	42.2
	SD	-----	1.5	0.5	2.1
PPS/40NiGr	AVE	-----	262.7	275.5	35.2
	SD	-----	2.1	1.3	1.3
PPS/30NiGr/ 20NiFlk	AVE	-----	261.6	276.4	29.0
	SD	-----	2.1	0.1	3.5
PPS/30NiGr/15SS	AVE	-----	262.1	276.4	34.3
	SD	-----	2.4	0.1	1.6
PPS/20NiGr/30Gr	AVE	-----	262.1	278.8	31.7
	SD	-----	0.4	0.4	0.1
PPS/40NiGr/ 15NiFlk	AVE	-----	265.2	279.1	19.4
	SD	-----	3.4	0.4	2.0
PPS/20Gr/15SS	AVE	-----	263.8	279.4	38.3
	SD	-----	0.9	0.8	2.8
PPS/25NiGr/10SS	AVE	-----	263.2	279.5	38.0
	SD	-----	1.8	1.6	2.1
PPS/10NiGr/25Gr	AVE	-----	265.2	279.6	31.8
	SD	-----	2.5	0.3	0.3
PPS/25NiGr/10Gr	AVE	-----	266.8	280.8	31.2
	SD	-----	3.9	1.2	3.2
PPS/40Gr	AVE	-----	265.8	281.7	27.1
	SD	-----	1.1	0.1	0.6
PEEK/Unfilled	AVE	130.6	329.3	339.8	32.1
	SD	0.8	7.4	1.6	0.6
PEEK/25NiGr/ 10SS	AVE	129.3	317.8	337.2	37.5
	SD	34.0	3.7	2.3	5.4
PEEK/30NiGr/ 20NiFlk	AVE	136.0	323.3	341.9	17.9
	SD	3.3	0.1	<0.1	0.5
PEEK/40NiGr	AVE	138.4	326.1	343.1	21.6
	SD	2.8	2.2	1.1	8.6
LCP300/Unfilled	AVE	117.8	411.4	423.3	3.0
	SD	0.8	0.4	1.1	1.2
LCP300/30Gr	AVE	127.8	-----	-----	-----
	SD	3.8	-----	-----	-----
LCP500/Unfilled	AVE	123.4	425.7	443.0	3.5
	SD	0.6	36.8	42.8	0.6

Figure 12. Specific Heats of Unfilled Polymers



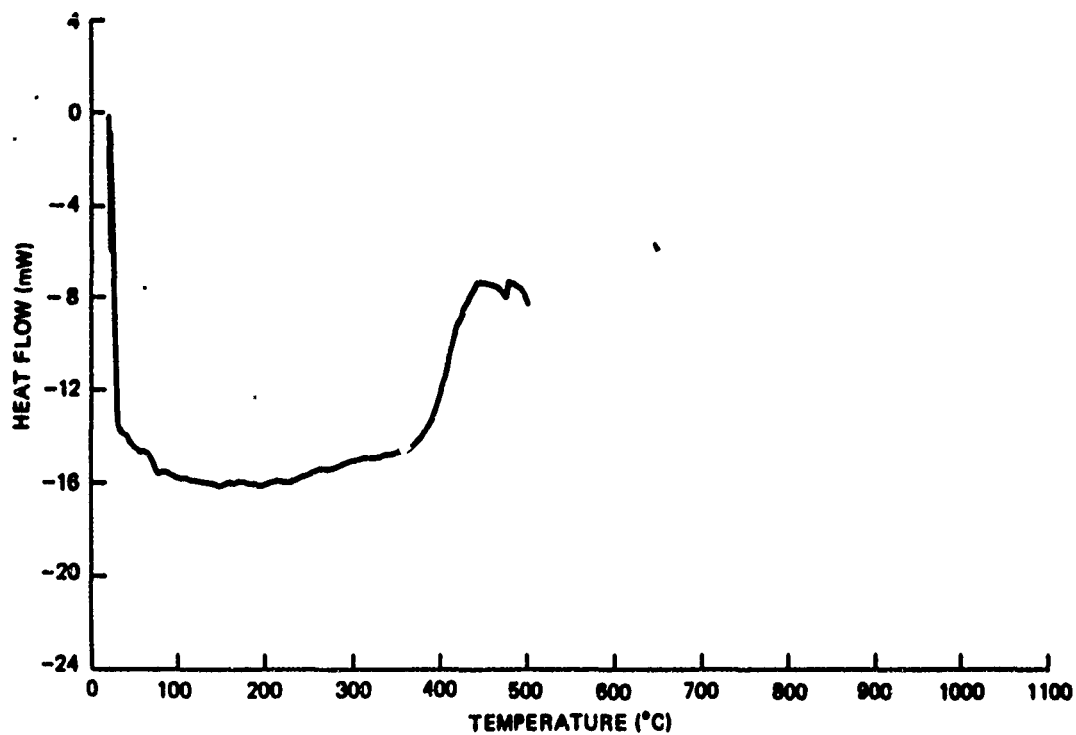


Figure 13. DSC Curve of LCP300/30%Gr

SUMMARY

Various conductive reinforcements in high temperature thermoplastic matrices were injection molded to formulate unique hybridized conductive composites. Concentrations of 25% to 30%NiGr fibers in combination with 10% to 15%SS fibers in PPS provided at least 80 dB of shielding at 80, 100, 150, 200 and 300 MHz and had electrical resistivities as low as 0.60 ohm-cm. The performance of PPS/30%NiGr/20%NiFlk was comparable to the highly loaded PPS/NiGr/SS combinations. Optical microscopy revealed that the PEEK composites had lower void contents than PPS composites with similar loadings. Additionally, the PPS composites loaded with NiGr/SS combinations appeared to contain the largest voids. Generally, PEEK composites had better mechanical properties than PPS composites. The unfilled PEEK resin displayed ductile failure at both test temperatures while PPS failed in a brittle manner at room temperature and in a ductile manner at 104°C. PEEK composites yielded the highest tensile strengths (156.5 MPa for PEEK/40%NiGr). PEEK/25%NiGr/10%SS exhibited the highest flexural strength at room temperature (238.6 MPa) while PEEK/40%NiGr had the highest value (199.9 MPa) at 104°C. The unfilled PEEK absorbed the most energy (>120 joules) during impact testing. The highest value for a reinforced thermoplastic was 9.10 joules for PEEK/25%NiGr/10%SS.

All of the materials absorbed less than one percent by weight of water except for PPS/40%NiGr/15%NiFlk (1.404%) and PPS/30%NiGr/10%SS (1.151%). Density values ranged from 1.98 g/cc for PPS/30%NiGr/20%NiFlk to 1.47 g/cc for LCP300/30%Gr. Differences between calculated and measured densities were attributed to voids present in the composites. The glass transition temperatures of unfilled PPS, PEEK, LCP300 and LCP500 were 88.8°C, 130.6°C, 117.8°C and 123.4°C, respectively. The neat PPS resin was 47.5 percent crystalline while the PEEK polymer was 24.7 percent crystalline.

The primary objective of this investigation was to develop conductive composites with enhanced electrical properties as compared to conventional single reinforcement composites (i.e., PPS/40%NiGr). By utilizing a hybridized reinforcement approach, several materials provided these properties. These advanced conductive composites offer significant weight savings, lower processing costs, improved corrosion resistance, reduced maintenance costs and better reliability compared to existing aluminum avionics components. In conclusion, this characterization study provides a foundation for the selection of non-metallic materials to replace metals in non-structural, EMI critical aircraft applications.

FUTURE EFFORTS

The major thrust of future work should be spent on implementing these conductive composites into specific avionic components such as connectors, avionic housings, waveguides, etc. Pursuing aircraft applications for these materials should demonstrate the feasibility of this approach to solving fleet EMI and corrosion problems simultaneously.

Additionally, processes such as thermoforming and stamp molding should be investigated to improve on the EMI shielding and toughness characteristics of advanced conductive composites. Metallic coatings deposited on the outer surfaces of reinforced polymeric systems should be studied to improve contact resistance properties. Additional work should be performed using new conductive materials such as nickel fibers, intercalated graphite powders and fibers and silver plated nickel spheres in hybrid reinforcement schemes. New matrix materials with good resistance to environmental and solvent deterioration along with enhanced toughness characteristics should also continue to be investigated.

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